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GUIDELINES FOR METHODS TO BE ADOPTED FOR IDENTIFYING THE MINEROLOGY OF IRON OXIDES; LUMP ORES, SINTERS AND PELLETS

UDC 622·341·1· 188·5 : 543·063

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 2

June 1987

Indian Standard

GUIDELINES FOR METHODS TO BE ADOPTED FOR IDENTIFYING THE MINEROLOGY OF IRON OXIDES; LUMP ORES, SINTERS AND PELLETS

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Indian Standard

GUIDELINES FOR METHODS TO BE ADOPTED FOR IDENTIFYING THE MINEROLOGY OF IRON OXIDES; LUMP ORES, SINTERS AND PELLETS

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 1 December 1986, after the draft finalized by the Ores and Raw Materials Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 In order to fully understand the mechanism of sintering, the phases involved in bonding the sinter, the role of gangue materials in determining the quality of sinter as well as for general assessment of sinter quality, it is necessary to identify under a microscope, the various phases present in any sinter. To facilitate the identification of these phases, these guidelines have been formulated.

0.3 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places ratained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the method for identifying the major constituents present in the microstructures of iron oxides; lump ores, sinter and pellets.

2. TERMINOLOGY

2.1 The principal constituents present in the microstructure of iron ore, sinter and pellets taken for the purpose of identification shall include:

a) Haematite as Fe_2O_3

b) Magnetite as Fe_3O_4

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*Rules for rounding off numerical values (*revised*).

c) Wustite as FeO

d) Fayalite as Fe_2SiO_4

e) Calcium ferrite*

3. TEST SAMPLE

3.1 The samples chosen for the minerological identification should be representative. At least 5 samples for each of the iron bearing materials of 10-15 mm size should be taken for this purpose.

4. METHODS FOR IDENTIFICATION

4.1 The following combined methods are applied for identification of minerology:

a) Identification by microscopic observation,

b) Identification by Etch test, and

c) Identification by microhardness measurement.

4.1.1 *Identification by Microscopic Observation* — The following two methods may be adopted for identification by microscopic observation:

a) Reflectance method, and

b) Transmission method.

The former is adequate for simple identification.

4.1.1.1 *Sample for preparation for reflection type microscopic examination* — The sample for reflection type microscopic examination should be prepared either by mounting in resin or used as it is before polishing but reduced materials are preferably mounted in resin. The polishing should be done in a normal metallographic sample polishing wheel starting from coarse emery to fine emergy paper. Final polishing should be done by a buffing cloth wheel using corrundum paste. However, it is always preferable to use a diamond polishing wheel with diamond paste at the final stages of polishing. The entire polishing should be done by hand and care should be taken to see that no small pieces get chipped off from the surface during polishing.

4.1.1.2 *Preparation of the laminate samples for transmission microscope* — The laminate sample for transmission microscopy should be ground so that the surfaces are parallel to each other. One of the ground surfaces should then be carefully polished on a glass plate with Canada Balsom as the polishing agent. The glued sample has to be polished until it becomes 0·03 mm thick and then, it has to be covered with the slide glass.

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*Calcium ferrites are classified as follows:

1. Calcium ferrite in binary system $CaO.2Fe_2O_3$, $CaO.Fe_2O_3$, $2CaO.Fe_2O_3$
2. Calcium ferrite in ternary system
3. $CaO.FeO.7Fe_2O_3$, $CaO.FeO.Fe_2O_3$, $4CaO.FeO.4Fe_2O_3$ and $CaO.3FeO.Fe_2O_3$

4.2 Identification by Etch Test — The sample used for the reflection microscopy is immersed into an etching solution contained in a small container like an evaporating dish, after the microscopic observation. The sample is kept in etching solution for a certain period of time, taken out from the solution, washed under running water or with alcohol, dried by blowing warm air and then subjected for the microscopic observation.

4.3 Identification by Microhardness Measurement — The microhardness measurement are carried out as a supplement to phase identification by reflection microscopy. The sample used for reflection microscopy itself is used as the sample for microhardness measurement. Microhardness is measured by the size of deformation which is traced with a diamond drill under a load of about 100-200 gm. The structure is then interpreted on the basis of the microhardness obtained. The sample used for the microhardness measurement should be chosen from the reflectance samples only if relatively less amount of cracks are present.

5. PROCEDURE

5.1 For the purpose of identification of the major minerological constituents present in iron ore, sinter and pellets, the above procedure shall apply. The major phases to be identified in accordance with various properties have been given in Tables 1 and 2.

TABLE 1 PROPERTIES OF MINERALS PRESENT IN IRON ORE, SINTERS AND PELLETS

(*Clause* 5.1)

SL No.	MINERALS	CRYSTAL PROPERTIES	OPTICAL PROPERTIES	ETCHING PROPERTIES	MICRO-HARDNESS
(1)	(2)	(3)	(4)	(5)	(6)
1.	Haematite (Fe_2O_3)	Hexagonal system	Refractive Index (R.I.) = 3·22. Thick greyish white layer with bluish tint. Reflection brightness is very high re-oxidised haematite — needle like platy, or polygonal structure under transmitted light — Reddish brown colour	Cannot be etched by Aqua-Regia (3 parts HCl : 1 part HNO_3)	1 000 HV
2.	Magnetite (Fe_3O_4)	Cubic system	Refractive Index (R.I.) = 2·42. Thick grey layer with brownish tint reflection brightness is low. Massive crystals of irregular shape often crystallised in the form or dendrites, tetrahedron or octahedron opaque under transmitted light	Turns yellow in Aqua Regia	600-700 HV
3.	Wustite (FeO)	Cubic system	Refractive Index (R.I.) is lower than Fe_3O_4. Little darker in colour than Fe_3O_4. Exists in granular or dendritic forms and often observed as the form of eutectic with Fe_3O_4 Opaque under transmitted light	Etched completely in an aqueous solution saturated with $Sncl_2$ for about 1 to 2 minutes exposure	450-500 HV
4.	Fayalite (Fe_2SiO_4)	Orthorombic system	Refractive Index (R.I.) = 1·886. Crystallise in the form of long and lathy type crystales. Under transmitted light, colour less in parallel nichol but light yellow, yellow green or orange colour in crossed nichols	—	600-700 HV
5.	Calcium Ferrite	Rectangular	Optically anisotrapic. General crystal form is rectangular but ocassionally crystallised in irregular shape-massive, needle like or dendritic form. Grey in colour under reflected light. Reflectivity higher than silicate in matrix but lower than magnetite. Reddish orange under transmitted light in parallel nichols. Rectangular crystals show straight distinction	Etching properties depend on state of combination between iron oxide and lime (*see* Table 2)	800-900 HV

TABLE 2 ETCHING BEHAVIOUR OF CALCIUM FERRITE MINERALS

(*Clause* 5.1)

SL No.	MINERAL	ETCHING TIME (SEC)		
		20 Percent HCl Soln. 21°C	20 Percent HCl Slon. 60°C	50 Percent HCl Soln. 60°C
(1)	(2)	(3)	(4)	(5)
1.	$2CaO.Fe_2O_3$	15-30	—	—
2.	$CaO.FeO.Fe_2O_3$	No etch	15-60	—
3.	$4CaO.FeO.4Fe_2O_3$	,,	15-60	—
4.	$3CaO.FeO.7Fe_2O_3$	,,	No etch	20-30
5.	$CaO.2Fe_2O_3$*	,,	,,	Slightly etch
6.	$CaO.Fe_2O_3$	,,	,,	Very slightly etch

*The presence of mineral is confirmed by etching consisting of a 60 to 120 Sec exposure to 10 percent HF, 40 percent water and 50 percent alcohol solution after the etch in 50 percent HCl at 60°C.

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